IS 4589 (2002): Plastic Clay and Washed Plastic Clay for Ceramic Industry [CHD 9: Ceramicware]

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भारतीय मानक

सिरामिक उद्योग के लिए प्लास्टिक मिट्टी और प्रक्षलित प्लास्टिक मिट्टी — विशिष्टि

(तीसरा पुनरीक्षण)

Indian Standard

PLASTIC CLAY AND WASHED PLASTIC CLAY FOR CERAMIC INDUSTRY — SPECIFICATION

(Third Revision)

ICS 81.060.10

BUREAU OF INDIAN STANDARDS
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September 2002 **Price Group 7**

Ceramicware Sectional Committee, CHD 9

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Ceramicware Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1968 under the title 'Ball clays for ceramic industry'. However, as the resources of ball clays, corresponding to the requirements of the standard are very limited, the ceramic industry has to use ball clay of a slightly inferior grade and in many cases has to resort to silicious plastic clays after suitable pretreatment. Keeping this aspect in view, the standard was revised in 1979 and in the first revision, both plastic clay and ball clay were covered. In the second revision, washed plastic clay as Type 2 was also included because of its use in ceramic industry.

In this revision, the requirement of water of plasticity has been modified in the light of experience gained. Moreover, the methods of test for various characteristics have been given in greater detail apart from rectifying any printing errors.

The composition of the committee responsible for the formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PLASTIC CLAY AND WASHED PLASTIC CLAY FOR CERAMIC INDUSTRY — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirement, and methods of sampling and test for plastic clays for use in ceramic industry.

2 REFERENCES

The following Indian Standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
460 (Part 1) : 1985	Specification for test sieves : Part 1 Wire cloth sieves (*third revision*)
1070 : 1992	Reagent grade water (*third revision*)
2781 : 1975	Glossary of terms relating to ceramicware (*first revision*)
2837 (Part 2) : 1977	Poreclain crucibles and basins : Part 2 Basins (*first revision*)

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 2781 shall apply.

4 TYPES AND GRADES

4.1 The material shall be of the following two types:

a) *Type* 1 — Unwashed plastic clay, and
b) *Type* 2 — Washed plastic clay

4.1.1 Type 1 shall have three grades, namely, Grade 1, Grade 2 and Grade 3.

5 REQUIREMENTS

5.1 Material

The material shall be in the form of lumps or powder free from foreign matter and shall normally slake in water. However, some plastic fire clays are available in hard lump form and do not slake easily in water.

5.2 The material shall also comply with the requirements of Table 1 when tested in accordance with methods given in Annex A.

6 PACKING AND MARKING

6.1 Packing

The material shall be packed in jute bags or as agreed to between the purchaser and the supplier.

6.2 Marking

The bags shall be securely stitched and marked indelibly with the following information:

a) Name and grade of the material,
b) Indication of the source of manufacture, and
c) Net mass in kg.

7 SAMPLING

Representative samples of the material shall be drawn and criteria for conformity shall be determined in accordance with the procedure prescribed in Annex B.

Table 1 Requirements for Plastic Clays and Washed Plastic Clays for Use in Ceramic Industry

(*Clause* 5.2)

Sl No.	Characteristics	Requirement				Method of Test (Ref to Cl No. of Annex A)
		Type 1			Type 2	
		Grade 1	Grade 2	Grade 3		
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Grit content (residue on 45-micron IS sieve), percent by mass, *Max*	2.0	3.0	4.0	1.0	A-1
ii)	Particle size distribution, percent by mass:					
	a) Coarser than 25 microns, *Max*	3	6	9	0.1	
	b) Finer than 10 microns, *Min*	90	88	85	95	A-2
	c) Finer than 2 microns, *Min*	70	65	45	75	
iii)	Plasticity by hand feel test	Very good	Good	Fair	Very good	–
iv)	Water of plasticity, percent by mass, *Min*	34	32	30	35	A-3
v)	Atterberg number, *Min*	18	15	12	20	A-4
vi)	Loss on ignition, percent by mass, *Min*	10.5	9.5	9.0	11.0	A-5
vii)	Alumina (as Al_2O_3), percent by mass, *Min*	28	25	20	28	A-6
viii)	Oxide of iron (as Fe_2O_3), percent by mass, *Max*	1.5	2.0	2.0	1.0	A-7
ix)	Titania (as TiO_2), percent by mass, *Max*	1.5	2.0	2.0	1.5	A-8
x)	Iron oxides and titania combined (as $Fe_2O_3 + TiO_2$), percent by mass, *Max*	2.75	3.5	3.75	2.5	A-7 and 8
xi)	Linear shrinkage (dry at 110°C), percent, *Max*	6	7	8	6	A-9
xii)	Fired colour at 1 350°C	Creamish white	Pale cream to dull cream	Pale cream to dull cream or light grey	White to pale cream, should be free from specks	A-10
xiii)	Maturity (water absorption) on firing at 1 350°C, *Max*	3	5	9	2	A-10
xiv)	Total fired linear shrinkage (at 1 350°C), *Max*	15	13	12	16	A-11
xv)	Modulus of rupture, Mpa (on dry specimens, dried at 110°C), *Min*	3.5	3.0	2.5	4.0	A-12

ANNEX A

(*Clause* 5.2)

A-0 GENERAL

A-0.1 Test for physical and chemical requirements on plastic clays shall be carried out on clay samples as prepared in **B-5**.

A-0.2 Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

A-1 DETERMINATION OF GRIT CONTENT

A-1.0 Outline of the Method

Clay is crushed to pass a 250-micron IS sieve and deflocculated in water using sodium carbonate. The slurry is then sieved through a 45-micron IS sieve and the residue retained on it is reported as grit.

A-1.1 Apparatus

A-1.1.1 *Balance*

A physical balance sensitive to 0.1 g and an analytical balance sensitive to 0.001 g.

A-1.1.2 *Mechanical Stirrer*

The stirrer shall essentially be a mechanically operated stirring device in which a suitably mounted electric motor turns a vertical shaft at a speed of not less than 1 000 rev/min without load. The shaft shall be fitted with a replaceable stirring paddle made of non-rusting metal, plastics or hard rubber. A special dispersion cup, fitted with wire baffles, shall be provided to hold the sample.

A-1.1.3 *Sieves*

250-micron, 45-micron IS sieves [*see* IS 460 (Part 1)].

A-1.2 Preparation of Sample

Crush the clay sample to pass completely through a 250-micron IS sieve. Place in an air oven, dry at 105 to 110°C for not less than 5 h and then cool in a desiccator.

A-1.3 Procedure

Weigh about 250 g of the sample prepared in **A-1.2** to the nearest 0.1 g and transfer to a glass container of 2-litres capacity. Wet the clay sample with 1 000 ml of distilled water, containing sodium carbonate, 0.2 percent by mass of the sample taken for the test. Allow to slake for 24 h and then blunge with the mechanical stirrer for 30 min.

A-1.3.1 Transfer the mixture to the 45-micron IS sieve and wash it through the sieve with a gentle flow of water from a rubber hose until the water passing through the sieve appears to be clear and free from cloudiness. Take due care to prevent any loss of the sample because of splashing or overflowing. Dry the sieve with residue at 110 ± 5°C for 2 h.

A-1.3.2 Carefully brush the residue on to a tared watch glass and weigh to the nearest 0.001 g.

A-1.4 Calculation

$$\text{Grit content, percent by mass} = \frac{M_1 \times 100}{M}$$

where

M_1 = mass in g of the residue, and
M = mass in g of the prepared sample (**A-1.3**) taken for the test.

A-1.4.1 Report the average result of three specimens. Result of duplicate tests on each specimen shall agree within 0.5 percent by mass of the sample taken for the test. If results of duplicate tests do not agree within the tolerance limit, they shall be rejected and two additional tests shall be carried out.

A-2 DETERMINATION OF PARTICLE SIZE DISTRIBUTION

A-2.0 Principle

The progress of sedimentation of clay suspension in a tall cylinder is followed by withdrawing sample portions at a known depth, based on the temperature of the suspension below the surface with a pipette and weighing the sediment. Stoke's law is then applied to find out grain size which is expressed as the width of a cube.

A-2.1 Apparatus

A-2.1.1 *Andreasen's Pipette* — The pipette (*see* Fig. 1) consists of the following:

a) *Sedimentation cylinder* — A glass sedimentation vessel (*W*) with about 5.3-5.4 cm internal diameter, having a graduated scale 0 to 20 cm with sub-graduation in mm or 0-200 mm marked on the side of the vessel. The zero mark of the scale should be 4-5 cm but not less than 4.0 cm from the inside base of the vessel. The capacity of the vessel, after inserting the pipette (*P*) in the vessel (*W*) and, when filled with water up to the 20-cm mark, should be exactly 550 ml with three-way cock in open position. The vessel is provided with a neck to take a pipette through a ground-glass stopper.
b) *Pipette* — The pipette has a bulb (*see* Fig. 1) and is provided with a two-way tap and side discharge tube. The capacity of the pipette up to the graduation line, when the tap is in a 'closed' position, is 10 ml. A bell-shaped dome is fused to the capillary stem of the pipette below the tap to provide a ground-glass joint to fit the neck of the sedimentation vessel. A small vent hole is made in the dome to avoid creation of low pressure in the pipette during successive sampling. The tip of the stem, when the pipette is fitted to the vessel, shall be level with the zero mark of the scale. The stem of the pipette from the bulb to the two-way stopcock and after is constructed of capillary glass tube having a bore diameter between 1.0 mm and 1.3 mm. The tube above the bulb should have a bore diameter of 4.0 to 4.5 mm.

A-2.1.2 *Basin*

Silica or porcelain basin [*see* IS 2837 (Part 2)].

A-2.2 Reagents

A-2.2.1 *Sodium Pyrophosphate Solution*

Dissolve 24.5 g of sodium pyrophosphate ($Na_4P_2O_7.10H_2O$) in 500 ml of distilled water.

A-2.3 Procedure

A-2.3.1 *Preparation of Sample*

Crush the clay sample to pass completely through a 250-micron IS sieve. Dry a portion of the sample (~ 15 g) at 110 ± 2°C to constant mass.

A-2.3.2 Place 5.50 g of dried clay sample in a 250-ml wide mouth bottle and add 200 ml of distilled water. Allow to stand for 24 h. Add 10 ml of sodium pyrophosphate solution. Agitate the bottle with its contents by rotating end-over-end for 17 h.

NOTE — This is best done by packing the bottle in the jar of a pot mill so as to turn end-over-end as the mill rotates.

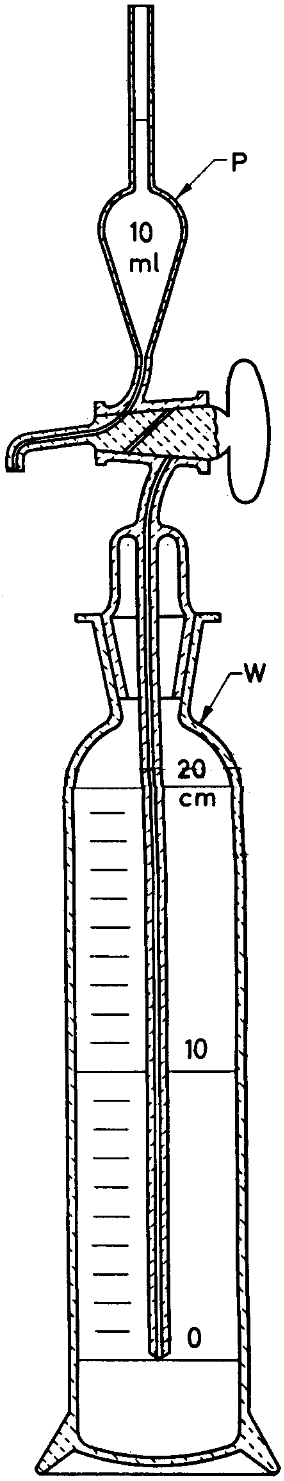

FIG. 1 ANDREASEN'S PIPETTE

A-2.3.3 Transfer the dispersed sample to the Andreasen's pipette and first add distilled water to bring the level of the suspension at about 18-cm mark on the scale and then insert the pipette in the vessel with stopcock in an open position and then bring the level of the suspension up to 20-cm mark on the scale by adding distilled water, making up the total volume to 550 ml.

NOTE — This produces a 1 percent suspension and the concentration of the elctrolyte is 0.002 g mole ($Na_4P_2O_7 . 10H_2O$) per litre.

A-2.3.4 Place the Andreasen's pipette in a water bath maintained at a constant temperature (*see* Table 2) for at least 30 minutes to permit it to attain the temperature of the bath. Remove it, shake by hand for two minutes and replace immediately into the bath. Hold the pipette for about 30 s to attain stability and then start counting of time by a stop-watch for the initial period. Withdraw 10 ml of suspension at increasing intervals of time, at a uniform rate of a cut 20 s as withdrawal time. The samples are drawn at intervals of 2, 5, 10 and 30 minutes and then at 1, 2, 4, 6 and 24 h.

NOTE — These intervals are chosen arbitrarily, but should be adhered to in order to save time in calculating the grain size.

A-2.3.5 Take 10-ml aliquots in small, weighed basins, and evaporate to dryness at 110°C. Weigh the residue accurately to 0.1 mg.

A-2.4 Calculation

Calculate the percentage of net dried residue making a correction for the sodium pyrophosphate content in the mass of the dried residue by subtracting 0.005 3 g as follows:

$$\text{Net dried residue, percent by mass, } M_1 = \frac{(M_1 - 0.005\ 3)}{M_2} \times 100$$

where

M_1 = mass of the dried residue , and

M_2 = theoretical mass of the clay sample in 10 ml of suspension.

Table 2 Values of Viscosity and Density of Water

(*Clauses* A-2.3.4 *and* A-2.5.1)

Sl No.	Temperature	Viscosity of Water, η	Density of Water D
(1)	(2)	(3)	(4)
	°C	g/cm.s	g/cm³
i)	10	0.013 10	0.999 7
ii)	20	0.010 09	0.998 2
iii)	25	0.008 95	0.997 0
iv)	27	0.008 55	0.996 5
v)	30	0.008 00	0.995 6
vi)	40	0.006 56	0.992 3

A-2.5 Calculation of Particle Size Distribution

The particle size distribution is calculated by applying the Stoke's law which may be stated as follows:

$$r = \sqrt{\frac{9h\eta}{2(D_1 - D_2)gt}} \qquad \ldots (1)$$

where

r = radius of spherical particle in cm,

h = height in cm of the liquid surface from the tip of the pipette before the suspension is withdrawn,

η = viscosity in g/cm.s of the suspending medium at the temperature of the test

D_1 = density in g/cm^3 of the solid particle,

D_2 = density in g/cm^3 of the suspending medium at the temperature of test,

g = acceleration due to gravity (979 cm/s^2), and

t = time in seconds from the start of the test.

For a solid suspended in a liquid medium, the fraction $9\eta/2\,(D_1-D_2)$ g is constant at a given temperature and can be evaluated. Thus, equation (1) may be expressed as follows:

$$r = k\sqrt{\frac{h}{t}} \qquad \ldots (2)$$

where

$$k = \sqrt{\frac{9\eta}{2(D_1 - D_2)g}} \qquad \ldots (3)$$

A-2.5.1 Substituting in equation (3) the values for:

a) density of clay, D_1 = 2.625 g/cm^3 approx. (Determine the specific gravity of the clay sample by using psychrometer as per the standard procedure);

b) acceleration due to gravity, g = 979 cm/s^2 (*see* **A-2.5**);

c) viscosity of suspending medium (water) at 40°C, η = 0.006 56 g/cm.s (*see* Table 2); and

d) Density of suspending medium (water) at 40°C, D_2 = 0.992 3 g/cm^3 (*see* Table 2).

We have the value of constant k = 0.004 297. Applying this value of k in equation (2) and multiplying by 10^4, we get the value of r in microns.

A-2.5.2 Stoke's law is applicable to angular and cubical particles of the same mass as spherical particles. For calculating the grain size as width (or edge length) of a cube of the same volume as sphere of radius r, the width of the cube may be expressed as follows:

Width of the cube, C= 1.612 r

NOTE — This width of the cube shall be adopted for expressing the results of test for particle size distribution in a clay.

A-2.5.3 Determine the radius 'r' of the particles of the clay sample by using equation (2) (*see* **A-2.5**), and appropriate values for viscosity and density of water at the temperature of test (*see* Table 3) and convert into width of cube (*see* **A-2.5.2**) and plot a smooth graph with C as the abscissa and percentage of mass M (*see* **A-2.4**) finer than the corresponding size as the ordinate. Finally, construct a histogram to find out the particle size distribution for the specified size ranges. A model calculation of particle size distribution is illustrated in Table 3, and a graph and histogram based on data given in it are drawn in Fig. 2 for guidance.

Table 3 Model Calculation for the Determination of Particle Size Distribution of a Clay

(*Clause* A-2.5.3)

Time of Drawing Sample from Start of the Test	Radius of Particle	Width of the Cube C= 1.612 γ	Mass of Empty Dish	Mass of Empty Dish and Sample	Mass of Sample	Corrected [1)] Mass of Sample	Under Size
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
seconds	microns	microns	g	g	g	g	percent cumulative
120	17.54	28.27	35.026 2	35.128 6	0.102 4	0.097 1	96.5
300	10.98	17.70	38.065 9	38.167 0	0.101 1	0.095 8	95.8
600	7.69	12.40	39.151 6	39.252 5	0.100 9	0.095 6	95.6
1 800	4.39	7.08	41.106 3	41.199 7	0.093 4	0.088 1	88.1
3 600	3.07	4.95	37.026 5	37.115 7	0.089 2	0.083 9	83.9
7 200	2.15	3.47	21.049 5	21.132 4	0.082 9	0.077 6	77.6
14 400	1.50	2.42	25.223 5	25.298 8	0.075 3	0.070 0	70.0
21 600	1.21	1.95	20.461 2	20.532 0	0.070 8	0.065 5	65.5
86 400	0.60	0.97	40.013 6	40.017 2	0.057 6	0.052 3	52.3

[1)] Value after subtracting 0.005 3 g, mass of electrolyte in 10 ml of suspension (*see* **A-2.4**), whereas theoretical mass of sample in 10 ml of suspension = 0.10 g.

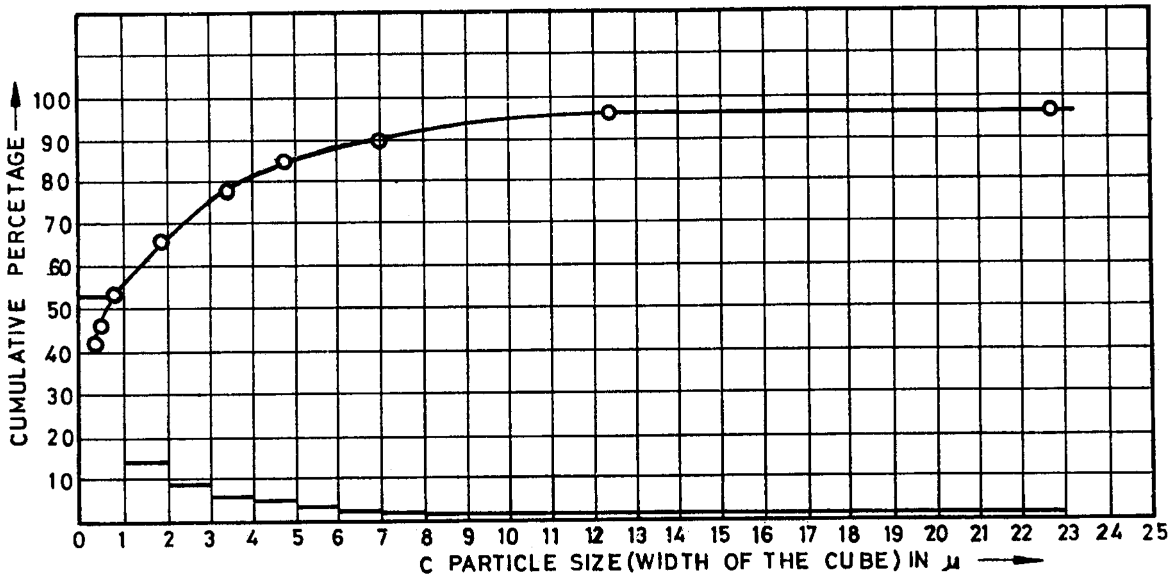

FIG. 2 MODEL GRAPH AND HISTOGRAM BASED ON DATA GIVEN IN TABLE 3

A-3 DETERMINATION OF WATER OF PLASTICITY

A-3.0 Outline of the Method

The water content of clay at the point of maximum workability is determined. This, when expressed as a percentage of oven-dried clay, gives the value for water of plasticity.

A-3.1 Apparatus

A-3.1.1 *Spatula*

A-3.1.2 *Porcelain Basin*

A-3.1.3 *Burette* — 50-ml capacity.

A-3.1.4 *Balance* — Accurate up to 0.01 g.

A-3.2 Procedure

Crush about 150 g of the clay sample (*see* **B-5**) to pass through a 250-micron IS sieve. Work up the sieved clay with distilled water, adding a small quantity at a time from a burette, to a soft plastic consistency and thoroughly wedge and knead by hand. Cover with a wet cloth and allow to age for 24 h. Again knead the plastic mass and pug, adding small quantity of water as necessary, until the mass attains working consistency for either extrusion or moulding into bars. Divide the mass into three equal portions rounding the edges and corners to prevent loss of material in handling. Place each portion on a separate tared watch glass and weigh immediately to the nearest 0.01 g.

A-3.2.1 Then dry them for 4 h at 70 ± 2°C and finally for 12 h and at 110 ± 5°C. Cool in a desiccator and weigh each to the nearest 0.01 g.

A-3.3 Calculation

$$\text{Water of plasticity, percent by mass} = \frac{M - M_1}{M_1} \times 100$$

where

M = mass in g of the plastic clay mass (**A-3.2**), and

M_1 = mass in g of the dried clay mass (**A-3.2.1**).

A-3.3.1 Report the average of three results. Results of test on each plastic mass shall agree within 0.5 percent by mass of the average value. If results of each test do not agree within the tolerance limit, they shall be rejected and the tests shall be repeated.

A-4 DETERMINATION OF ATTERBERG NUMBER

A-4.0 Principle

The difference in water contents between the lower limit of mobility (liquid limit) and the rolling limit (plastic limit) of the clay is determined. The value thus obtained is the Atterberg number of the clay.

A-4.1 Apparatus

A-4.1.1 *Porcelain Basin* — 110 mm in diameter.

A-4.1.2 *Spatula*

A spatula having a blade about 75 mm in length and about 20 mm in width.

A-4.1.3 *Liquid Limit Device*

A mechanical device consisting of a hard rubber base and a brass cup with carriage so constructed as to allow

the cup to be moved up and down by means of a crank. A sketch of the apparatus is given in Fig. 3.

A-4.1.4 *Grooving Tool*

Grooving tool of the shape shown in Fig. 4 . The blade of the tool shall have a width of 1 cm.

A-4.1.5 *Ground Glass Plate*

A-4.1.6 *Balance* — accurate to 0.01 g.

A-4.2 Determination of Liquid Limit

A-4.2.1 *Sample Preparation*

Crush about 250 g of the clay sample (*see* **B-5**) to pass through a 250-micron IS sieve and dry at 110 ± 5°C for 1 h.

A-4.2.2 *Adjustment of the Mechanical Device*

Adjust the height with the help of a gauge, 1 cm in

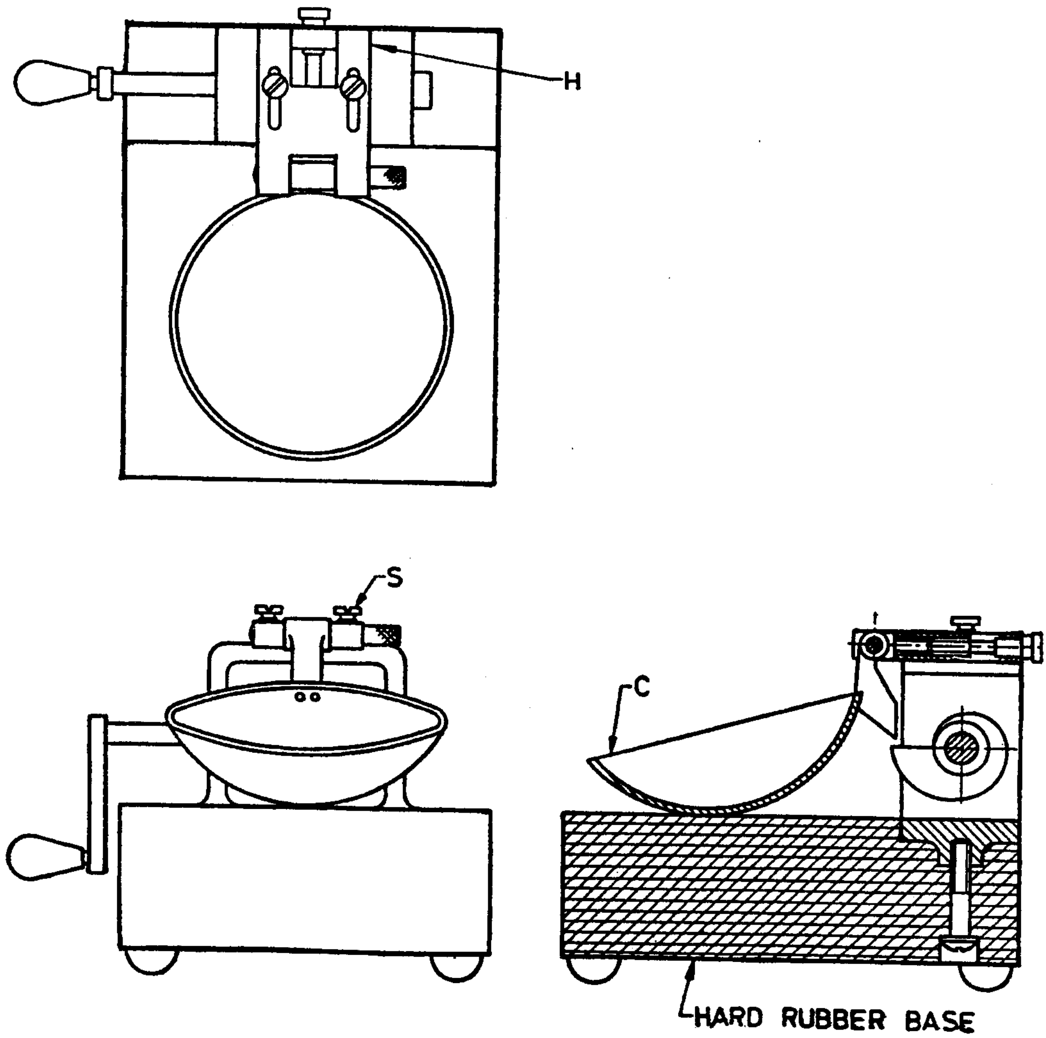

FIG. 3 MECHANICAL LIQUID LIMIT DEVICE

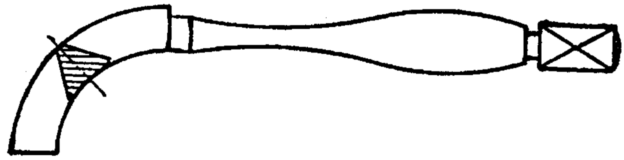

FIG. 4 GROOVING TOOL

width, to which the cup *C* is lifted so that the point on the cup that comes in contact with the base is exactly 1 cm above the base. Secure the adjustment plate *H* by tightening the screw *S*.

A-4.2.3 *Procedure*

Mix the clay sample (**A-4.2.1**) in the porcelain basin thoroughly with 10 to 15 ml of distilled water by kneading and chopping with a spatula. Add water further in increments of 1 to 3 ml. Thoroughly mix each increment of water before adding the next increment. When sufficient water has been added to produce a proper consistency, cover the plastic mass with a wet cloth and allow to age for 24 h. Again knead the plastic mass and then place the clay mass in the cup above the resting spot. Squeeze it down and spread it evenly with the spatula, care being taken to prevent entrapment of air bubbles. Level the clay mass with the spatula and at same time trim it to a depth of 1 cm at the point of maximum thickness. Return the trimmings to the basin. Divide the clay mass in the cup by firm strokes of the grooving tool along the diameter through the centre line of the cam follower so that a clean sharp groove of proper dimensions is formed. To avoid tearing of the sides of the grooves or slipping of the clay mass on the cup, up to six strokes, from front to back or from back to front, shall be permitted. Each stroke shall penetrate a little deeper until the last stroke from back to front scrapes the bottom of the cup clean. Make the groove with as few strokes as possible. Lift and drop the cup by turning the crank at the rate of two revolutions per second, until the two halves of the clay mass come in contact at any point along a distance of 1.25 cm on the line of the groove. Record the number of drops required to close the groove along a distance of 1.25 cm.

A-4.2.3.1 Remove a portion of the clay mass approximately of the width of the spatula that just flowed and place in a tared vessel. Weigh and then dry at 110 ± 5°C in an air oven and cool in a desiccator. Weigh after cooling. Record the loss in mass due to drying as the mass of water necessary to attain proper consistency.

A-4.2.3.2 Repeat the operation at least twice, adding more water each time to bring the clay to a more mobile condition. Thus, values above and below 25 are obtained for the number of drops required to close the groove.

A-4.2.4 *Calculation*

$$\text{Water content, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of water (**A-4.2.3.1**), and

M_2 = mass in g of the oven-dried clay.

A-4.2.4.1 *Flow curve* — Plot a flow curve on a semilogarithmic graph with the water contents as abscissa on the arithmetical scale and the corresponding number of strokes as ordinate on the logarithmic scale. The curve shall be a straight line drawn as nearly as possible through the three points.

A-4.2.4.2 *Liquid Limit* — Take the water content corresponding to the intersection of the flow curve with the 25-drop ordinate as the liquid limit of clay. Report the liquid limit value to the nearest whole number.

A-4.3 Determination of Plastic Limit

Crush about 10 g of the clay sample (*see* **B-5**) to pass through a 250-micron IS sieve and thoroughly mix with sufficient quantity of water to give a classic mass which can be easily shaped into a ball. Cover the plastic mass with a wet cloth and allow to age for 24 h. Again knead the plastic mass and then roll it between the fingers and the ground glass plate into a thread of uniform diameter throughout its length. The rate of rolling shall be 60 to 80 strokes per minute, counting a stroke as one complete motion of the hand forward and back to the starting position again. When the diameter of the thread becomes 0.32 cm, break the thread into six or eight pieces. Squeeze the piece together between the thumbs, knead and roll again. Continue this process of alternate rolling, gathering together, kneading and re-rolling, until the thread crumbles under the pressure required for rolling and cannot be rolled into a thread having a diameter of 0.32 cm. Weigh the crumbled pieces of the thread together. Dry them in an air oven at a temperature 110 ± 5°C, cool in a desiccator and weigh. Record the loss in mass and mass of the water necessary to attain that consistency.

A-4.3.1 *Plastic Limit*

Calculate the plastic limit, expressing the water content as percentage of oven dry clay, as follows:

$$\text{Plastic limit, percent} = \frac{(M_1 - M)}{M} \times 100$$

where

M_1 = mass in g of the crumbled pieces of thread, and

M = mass in g of the crumbled pieces of thread after drying.

A-4.4 Atterberg Number

Calculate the difference in water content of clay

between its liquid limit at 25 drops (obtained in **A-4.2.4.2**) and its plastic limit (obtained in **A-4.3.1**). This value thus obtained is the Atterberg number.

A-5 DETERMINATION OF LOSS ON IGNITION

A-5.1 Procedure

Take about 50 g of the material in china dish and heat to constant weight at 105 ± 2°C. Cool it in a desiccator and keep for subsequent tests.

A-5.2 Heat 1 g of the above dried material in a platinum crucible in a muffle furnace at 1 000 ± 50°C. Cool the crucible and put the contents in a desiccator to constant mass. The loss in mass denotes loss in ignition and the percent loss may be calculated.

A-6 DETERMINATION OF ALUMINA

A-6.0 Outline of the Method

Aluminium (also iron and titanium, when present) is complexed quantitatively with EDTA by boiling with an excess of the reagent. The excess EDTA is back titrated with standard zinc solution at *p*H about 5.2, using xylenol orange indicator. The aluminium EDTA complex (also titanium–EDTA complex, if present) on boiling with ammonium fluoride liberate EDTA which, when tirated with standard zinc solution, gives the amount of aluminium present in the solution. Due correction for titania is also carried out.

A-6.1 Reagent

A-6.1.1 *EDTA Solution* (0.025 M)

Dissolve 9.31 g of disodium salt of ethylene diamine tetra-acetic acid, dihydrate ($C_{10}H_{14}N_2Na_2O_8.2H_2O$) in water and dilute to 1 000 ml.

A-6.1.2 *Methyl Orange*

Dissolve 0.05 g of methyl orange in 100 ml of water.

A-6.1.3 *Dilute Ammonia* (1:5)

Dilute 25 ml of concentrated ammonia (R.D. 0.90) to 150 ml.

A-6.1.4 *Acetate Buffer* (*p*H 5.2)

Dissolve 21.5 g of sodium acetate ($CH_3COONa.3H_2O$) in 300 ml of water containing 2 ml of glacial acetic acid and dilute to one litre.

A-6.1.5 *Standard Aluminium Oxide Solution*

Weigh 0.529 3 g of aluminium metal (99.9 percent purity) into a beaker containing 40 ml of dilute hydrochloric acid (1:1) and small drop of mercury. Heat the beaker over a water bath. Transfer the solution to a 1 000-ml volumetric flask and make up to the mark. One millilitre of this solution is equivalent to 1.0 mg of aluminium oxide (as Al_2O_3).

NOTE — Mercury will not dissolve and may be removed while transfering the solution to 1 000-ml volumetric flask.

A-6.1.6 *Standard Zinc Solution* – 0.025 M.

Disslove 5.484 5 g of zinc acetate [Zn ($CH_3COOO)_2.2H_2O$] in 50 ml of glacial acetic acid and 25 ml of acetate buffer (**A-6.1.4**) and dilute to one litre in a volumetric flask. Find out the equivalent Al_2O_3 in g/ml from the procedure given in **A-6.2** taking 20 ml of standard aluminium solution (**A-6.1.5**).

A-6.1.7 *Xylenol Orange*

Dissolve 0.1 g of xylenol orange in 100 ml of water containing two drops of dilute hydrochloric acid (1:1).

A-6.1.8 *Ammonium Fluoride*

Dissolve 20 g of ammonium fluoride in 100 ml of water. Add 3 drops of xylenol orange (**A-6.1.7**) and then dilute hydrochloric acid (1 : 9) dropwise till the colour changes to yellow.

A-6.2 Preparation of the Sample Solution

Weigh accurately about 2 g of the dried material in a platinum dish (*see* **A-5.2**). Moisten the sample with 5 ml of water. Add 3 ml of dilute sulphuric acid and 20 ml of hydrofluoric acid and evaporate to dryness on the sand bath in a fume cupboard taking care to avoid spurting. Cool and repeat the process of evaporation twice with another 10 ml of hydrofluoric acid each time. Cool, add 2 ml of dilute sulphuric acid and evaporate as before. Heat the dry residue cautiously until fumes of sulphur trioxide cease. Ignite the residue at 1 000°C for 5 minutes and cool. Fuse the residue with 3 g of anhydrous sodium carbonate, cool the fused mass and dissolve it in 25 ml of dilute hydrochloric acid and dilute with water to 250 ml in a volumetric flask. Reserve the solution for determination of iron and titania.

A-6.3 Procedure

Transfer 100 ml of the solution prepared in **A-6.2** to a 500-ml conical flask. Add sufficient EDTA solution to provide an excess of at least 5 ml over the expected amount of alumina and iron (1 ml of 0.025 M EDTA = 1.25 mg Al_2O_3 approximately). Add 2 to 3 drops of methyl orange and then dilute ammonia dropwise until the colour changes from red to yellow. Add 5 ml of acetate buffer and about 25 ml of water and heat to boiling for 5 minutes. Cool, add 5 ml of buffer solution and titrate with standard zinc solution using 5 to 6 drops of xylenol orange indicator. The end point is indicated by a clear change from yellow to deep orange colour. Add 10 ml of ammonium fluoride solution and heat to boiling for 5 minutes.

Cool, add 5 ml of buffer solution and titrate the liberated EDTA with standard zinc solution.

NOTE — If titania content is above 0.5 percent, it may affect the sharpness of the end point.

A-6.4 Calculation

$$\text{Alumina, percent by mass} = \frac{(V \times F)}{M} \times 100 - 0.637\,8\,D$$

where

V = volume in ml of standard zinc solution required for the titration of liberated EDTA,

F = equivalent Al_2O_3 in g/ml of standard zinc solution,

M = mass in g of the sample represented by the aliquot taken in **A-6.3**, and

D = percent of TiO_2 present in the sample. The value having been obtained by photometric determination (*see* **A-8.3**).

A-7 DETERMINATION OF IRON OXIDE

A-7.0 Outline of the Method

The orange-red complex, produced by ferrous iron on addition of *o*-phenanthroline, is determined photometrically at approximately 510 nm.

A-7.1 Reagents

A-7.1.1 *Dilute Sulphuric Acid* — 1 : 1 (*v/v*).

A-7.1.2 *Hydrofluoric Acid* — 40 percent.

A-7.1.3 *Dilute Hydrochloric Acid* — 1 : 1 (*v/v*).

A-7.1.4 *Tartaric Acid Solution* — 10 percent (*m/v*) in water.

A-7.1.5 *Hydroxylamine Hydrochloride Solution* — Dissolve 1 g of the reagent in water and dilute to 100 ml.

A-7.1.6 *o-Phenanthroline Solution*

Dissolve 1 g of *o*-phenanthroline monohydrate in 90 ml of water with gentle heating and constant stirring. Cool and dilute to 100 ml with water.

A-7.1.7 *Acetate Buffer Solution*

Dissolve 21.5 g of sodium accetate (CH_3 COONa. $3H_2O$) in 300 ml of water containing 2 ml of glacial acetic acid and dilute to 1 000 ml with water.

A-7.1.8 *Standard Iron Solution*

Dissolve 7.02 g of ammonium ferrous sulphate [$(NH_4)_2SO_4$. $FeSO_4 6H_2O$)] in water containing 10 ml of concentrated sulphuric acid and dilute with water to 1 000 ml. Dilute 20 ml of this solution to 1 000 ml with water just before use. One millilitre of this diluted solution is equivalent to 0.02 mg of iron (as Fe).

A-7.2 Preparation of the Blank Solution

Prepare the blank solution following the procedure and using the same quantities of reagents given in **A-6.2** but without the material and dilute the solution with water to 250 ml in a volumetric flask.

A-7.3 Procedure

Transfer 5-ml aliquot of the sample solution prepared in **A-6.2** to a 50-ml volumetric flask. Add 2 ml of tartaric acid solution and 2 ml of hydroxylamine hydrochloride solution. Stir and add 5 ml of *o*-phenanthroline solution followed by 10 ml of acetate buffer solution. Mix the contents, allow to stand for 15 minutes and dilute with water up to the mark.

A-7.3.1 Transfer 0, 1, 2, 3, 4, 5, 6, 7, 8, 9 and 10 ml of the standard iron solution to eleven 50-ml volumetric flasks. Add 5 ml of the blank solution in place of the sample solution and the same quantities of reagents as in **A-7.3** to each of the volumetric flasks and dilute up to the mark with water.

A-7.3.2 Transfer suitable aliquots of the solutions prepared in **A-7.3.1** to the absorption cell and take photometric readings at 510 nm and plot a curve showing the amount of iron (as Fe) in mg against the readings. Next transfer an aliquot of the sample solution prepared in **A-7.3** to the absorption cell and record the photometric reading.

A-7.4 Calculation

Convert the reading obtained with the sample solution in **A-7.3.2** into milligrams of iron, using the calibration curve and calculate the percentage of iron oxide as follows:

$$\text{Iron oxide (as } Fe_2O_3\text{), percent by mass} = \frac{M}{M_1} \times 0.412\,9$$

where

M = mass in mg of iron found in the sample solution (**A-7.3.2**), and

M_1 = mass in g of the material represented by 5 ml of the aliquot taken in **A-7.3**.

A-8 DETERMINATION OF TITANIUM DIOXIDE (TiO_2)

A-8.0 Outline of the Method

The yellow coloured complex, produced in acid solution of the sample by the addition of hydrogen peroxide, is determined photometrically at approximately 410 nm.

A-8.1 Reagents

A-8.1.1 *Dilute Sulphuric Acid* — 1 : 1 (*v/v*).

A-8.1.2 *Phosphoric Acid*

Add 400 ml of phosphoric acid (R.D. 1.75) to 500 ml of water, cool and dilute to one litre.

A-8.1.3 *Hydrogen Peroxide* — 20 volumes.

A-8.1.4 *Standard Titanium Dioxide Solution*

Weigh accurately 3.680 g of potassium titanyl oxalate [$K_2TiO_2(C_2O_4)_2.2H_2O$]. Transfer to a boiling flask, add 8 g of ammonium sulphate and 100 ml of concentrated sulphuric acid. Gradually heat the mixture to boiling and boil for 10 minutes. Cool, pour the solution into 750 ml of water, and dilute to 1 000 ml in a volumetric flask. One millilitre of this solution is equivalent to 0.5 mg of titanium dioxide (as TiO_2).

A-8.2 Procedure

Transfer 25 ml of the prepared solution (**A-6.2**) to a 100-ml volumetric flask. Add 2.5 ml of dilute sulphuric acid, 5 ml of phosphoric acid and 5 ml of hydrogen peroxide. Make up the volume to the mark with water and mix.

A-8.2.1 *Calibration Curve*

Transfer 25 ml of each of the blank solution (**A-7.2**) to eleven 100-ml volumetric flasks and add 0, 1, 2, 3, 4, 5, 6, 7, 8, 9 and 10 ml respectively of the standard titanium dioxide solution using the same quantity of reagents of procedure as in **A-8.2**. Make up the solution up to the mark. Take the required aliquots of these solutions in the absorption cell, one at a time, and plot a curve showing optical densities of solution at 410 nm against the amount of titanium dioxide in milligrams present in each.

A-8.2.2 Next take appropriate aliquot of the solution prepared in **A-8.2** in the absorption cell and measure its optical density at 410 nm and note from the calibration curve the amount of titanium dioxide in milligrams present in it.

A-8.3 Calculation

$$\text{Titanium dioxide, percent by mass} = \frac{M_1}{M} \times 0.1$$

where

M_1 = mass in mg of titanium dioxide content present in the aliquot taken for the test (**A-8.2.2**), and

M = mass in g of the material present in the aliquot taken for the test (**A-8.2**).

A-9 DETERMINATION OF DRY LINEAR SHRINKAGE

A-9.0 Outline of the Method

Dry linear shrinkage of a clay is determined by measuring the decrease in one dimension of a plastic mass upon drying and expressing it as percentage on plastic basis.

A-9.1 Apparatus

A-9.1.1 *Mould*

A collapsible brass mould with internal dimensions of 25 mm × 25 mm × 120 mm, suitable for forming the test specimen of that size.

A-9.1.2 *Divider*

A-9.1.3 *Diagonal Scale*

A-9.1.4 *Air Oven*

Provided with thermostat and capable of drying clay at 110 ± 5°C.

A-9.2 Procedure

Take a portion of clay as prepared in **A-3.2** and again knead thoroughly. Coat the inside of the brass mould evenly with a thin layer of petroleum jelly or kerosene oil or light machine oil. Press a lump of the plastic clay mass into the mould. Take care to avoid entrapping any air. Scrap off any excess clay with a straight-edged spatula from the top of the mould and finally wipe off any adhering clay. Make the surface of clay smooth and mark two reference points approximately 100 mm apart. Measure the distance between the two points accurately to second place of decimal with divider and diagonal scale. Prepare five such test specimens.

A-9.2.1 Carefully release the test specimens from the mould and allow to dry slowly under a wet cloth for 48 h. Wipe off the petroleum jelly adhering to the specimen. Air dry the specimen for another 48 h at room temperature, occasionally turning sides of the test specimens to expose new surfaces for evaporation.

A-9.2.2 Alternatively, prepare the test specimen by pugging two or three times the plastic mass as prepared in **A-3.2** in a de-airing pug mill and extrude clay specimens in the form of either cylindrical bar or rectangular bar having cross-sectional area of about 150 mm^2 and 150 mm in length. Place the extruded test specimens on a lightly oiled glass plate and then mark two reference points approximately 100 mm apart. Measure the distance between the two points accurately to second place of decimal with divider and diagonal scale. Prepare five such test specimens. Do not disturb the test specimens until they attain leather hard condition during drying.

A-9.2.3 Place the test specimens in the air oven and dry them at 110 ± 5°C for 24 h. Remove the test specimens from the oven and keep in a desiccator to cool. Measure accurately to second place of decimal the distance between the two reference points.

A-9.3 Calculation — Calculate the dry linear shrinkage as follows:

$$\text{Dry linear shrinkage, percent} = \frac{L_p - L_d}{L_p} \times 100$$

where

L_p = distance in mm between the two reference points on the plastic mass, and

L_d = distance in mm between the two reference points on the dry mass.

NOTE — Round off the calculated values to the second place of decimal.

A-9.3.1 Report the average of results of test for five test specimens. Result of test on each bar shall agree within ± 0.5 percent of the average value. If any results does not agree within the tolerance limits, it shall be rejected and the test shall be repeated.

A-10 DETERMINATION OF MATURITY

A-10.0 Outline of the Method

Discs of china clay are fired to a temperature of 1 350°C and the water absorption is determined by soaking the fired discs in water.

A-10.1 Apparatus

A-10.1.1 *Balance* — Accurate to 0.01 g.

A-10.1.2 *Air Oven*

Capable of maintaining a temperature of 105 to 110°C.

A-10.1.3 *Electric Muffle Furnace*

Capable of maintaining a temperature of 1 350°C.

A-10.2 Preparation of Test Sample

Crush about 500 g of the clay sample (*see* **B-5**) to pass through a 250-micron IS sieve. Work up the sieved clay with distilled water, adding a small quantity at a time from a burette to a soft plastic consistency and thoroughly wedge and knead by hand. Cover with a wet cloth and allow to age for 24 h. Again knead the plastic mass and pug adding small quantity of water, as necessary, until the mass attains consistency for moulding.

A-10.2.1 Prepare discs about 25 mm in diameter and 10 mm in thickness by pressing by hand the china clay mass prepared in **A-10.2** in steel moulds. Cover the discs with a wet cloth and allow to dry for 24 h at room temperature. Then dry them for 4 h at 70 ± 2°C and finally for 12 h at 110 ± 5°C.

A-10.3 Procedure

Place the test pieces prepared in **A-10.2.1** into the electric furnace. Raise the temperature at the rate of 75°C per hour till temperature reaches 1 350°C.

At this stage, stop raising the temperature and soak for 30 min maintaining the temperature at 1 350°C and finally switch off the furnace. Allow the furnace to cool to room temperature. Take the test pieces out and cool in a desiccator. Note the fired colour of the test specimens.

A-10.3.1 Weigh accurately five individual test pieces nearest to 0.05 g. Immerse them in distilled water in a beaker taking care that they do not touch each other and also do not touch the sides or bottom of the beaker. Boil for five hours and then allow to soak in the same water for 24 h keeping the pieces immersed all the time.

A-10.3.2 Next day, lightly blot each piece with moist linen or cotton cloth to remove excess water from their surface and immediately weigh to the nearest 0.05 g.

A-10.4 Calculation

Calculate water absorption as a measure of maturity as follows:

$$\text{Water absorption, percent by mass} = \frac{M_1 - M}{M} \times 100$$

where

M_1 = mass in g of the test piece soaked in water (**A-10.3.2**), and

M = mass in g of the dry test piece (**A-10.3.1**).

A-10.4.1 Report the average of results of test for five test pieces. Results of test on each test piece shall agree within 0.5 percent of the average value. If any result does not agree with the tolerance limits it shall be rejected and the test shall be repeated.

A-11 DETERMINATION OF TOTAL FIRED LINEAR SHRINKAGE

A-11.1 Principle

The decrease in one dimension of a clay specimen in plastic state, when dried and heated to the required temperature, is measured.

A-11.2 Procedure

Take five test specimens as prepared and duly marked in **A-9.2.1** to **A-9.2.3**. Place them in a suitable furnance, and heat to 1 350°C (**A-10.3**). Remove the test specimens and cool in a desiccator. Measure the

distance between the two reference points with a vernier calliper accurately to the second place of decimal.

A-11.3 Calculation

A-11.3.1 Calculate the total linear shrinkage as follows:

$$\text{Total fired linear shrinkage, percentage} = \frac{L_p - L_f}{L_p} \times 100$$

where

L_p = distance in mm between the reference points on the specimen in plastic state, and

L_f = distance in mm between the two reference points on the fired specimen.

NOTE — Round off to the nearest second place of decimal.

A-11.3.2 Report the average of five test specimens.

A-12 DETERMINATION OF DRY MODULUS OF RUPTURE

A-12.1 Outline of the Method

Modulus of rupture is determined using extruded cylindrical or rectangular bars made out of the clay and dried carefully to avoid warping, cracking, etc.

A-12.1.2 Apparatus

A-12.1.1.2 *Testing Machine*

Any suitable testing machine having the following essential features may be used:

a) Two steel knife-edges rounded to a radius of 6.35 mm or more and approximately 80 mm apart for supporting the test specimen; and
b) A steel knife-edge also rounded to a radius of 6.35 mm or more for applying load at midpoint between the two supporting knife-edges directly at a uniform rate of loading not exceeding 44 N/min.

A-12.2 Procedure

Crush about 5 kg of the clay sample (*see* **B-5**) to pass through a 250-micron IS sieve. Work up the sieved clay with distilled water to a soft plastic consistency and thoroughly wedge and knead by hand. Cover with a wet cloth and allow to age for 24 h. Again knead the plastic mass and pug in a de-airing pug mill adding small quantity of water, as necessary, until the mass attains working consistency for extrusion. Finally, prepare at least 10 cylindrical or rectangular specimens measuring cross-sectionally about 150 mm^2, the length being adjusted such that after drying, the ends extend on either side of the supporting knife-edges at least 10 mm.

NOTE — A vacuum of not less than 700 mm of mercury shall be maintained during pugging operation.

A-12.2.1 Place the extruded test specimens on a lightly oiled glass plate, cover with a wet cloth and allow to dry for 24 h at room temperature. Then dry for 4 h at 70 ± 2°C and finally for 24 h at 110 ± 5°C, and cool in a desiccator.

NOTE — Due precautions shall be observed in the forming and drying of the test specimens so that they are straight and do not show warpage greater than one percent of their overall length. The bars shall be checked from time to time during drying for straightness and before they stiffen corrective straightening may be obtained by rotating the bars so that drying occurs from another surface region. Bars having flaws, warpage or voids, shall be rejected.

A-12.2.2 Remove a test specimen from the desiccator and immediately place on the supporting knife-edges of the testing machine so that the ends extend on either side by at least 10 mm. Apply the load at right angles to the specimen and midway between the supporting knife-edges at a uniform rate of not exceeding 44 N min, until failure occurs. Note the load which breaks the test specimen, measure the diameter at the point of breaking nearest to 0.03 mm at least at three points around the circumference at the cross-section and calculate the average.

A-12.2.3 Carry out this test on at least 10 specimens.

A-12.3 Calculation

Calculate the modulus of rupture for each test specimen as follows:

a) For cylindrical specimen:

$$M = \frac{8\,PL}{\pi\, d^3}$$

b) For rectangular specimen:

$$M = \frac{1.5\,PL}{BD^2}$$

where

M = modulus of rupture in Mpa,
P = load causing rupture in N,
L = distance in mm between the supporting edges,
d = diameter in mm of the test specimen,
B = breadth in mm of the test specimen,and
D = depth (height) in mm of the test specimen.

A-12.3.1 Report the average of results of test for 10 test specimens. The result of test on each test specimen shall agree within ±5 percent of the average value of results. If any result does not agree within the tolerance limit, it shall be rejected and another test specimen shall be tested.

ANNEX B

(*Clauses* 7 *and* A-0.1)

SAMPLING OF PLASTIC CLAY FOR CERAMIC INDUSTRY

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 The sample shall be placed in clean, dry, air-tight glass or other suitable containers.

B-1.4 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.5 Each sample container shall be sealed air-tight with a stopper after filling and marked with full details of sample, the date of sampling and other identification particulars.

B-1.6 The sample shall be stored in such a manner that during storage the properties of the material do not get affected.

B-2 SCALE OF SAMPLING FOR MATERIAL PACKED IN BAGS

B-2.1 Lot

In a single consignment, all the material belonging to the same grade and coming from the same supplier or source, shall constitute a lot.

B-2.2 Sub-lot

For the purpose of sampling, all the material in a lot shall be divided into a suitable number of homogeneous sub-lots of approximately equal mass in accordance with col 1 and 2 of Table 4.

B-2.3 The number of bags to be sampled from each sub-lot is given in col 3 of Table 4. These bags shall be selected at random from the sub-lot. From each of these bags, a small but equal increment of material shall be taken.

B-2.4 All the increments from the sample bags in the same sub-lot shall be pooled together to constitute a gross sample so that there are as many gross samples as the sub-lots into which a lot has been divided.

Table 4 Scale of Sampling for Bags

(*Clauses* B-2.2 *and* B-2.3)

No. of Bags in the Lot	No. of Sub-lots	No. of Bags to be Sampled from Each Sub-lot
Up to 10	1	All
11 to 200	2	5
201 to 500	3	10
501 to 2 000	4	15
2 001 and above	5	20

B-3 SAMPLING FROM BULK SUPPLIERS

B-3.1 Lot

The part or whole of the material in the bulk, belonging to the same grade and coming from the same supplier or source, shall constitute a lot.

B-3.2 Sub-lot

For the purpose of sampling, the material in a lot shall be divided into a suitable number of homogeneous sub-lots of approximately equal mass in accordance with Table 5 by suitably marking the lines of demarcation on the surface of the bulk.

B-3.3 Sampling from bulk shall be carried out as far as possible when the material is in motion. For this purpose, the increment shall be drawn at regular intervals, depending upon the rate of flow of the material of the sub-lot and the number of increments as given in col 3 of Table 5.

B-3.4 All the increments from a sub-lot shall be pooled together to constitute a gross sample.

B-4 SAMPLING FROM WAGONS

B-4.1 Lot

In a single consignment, all the material belonging to the same grade and coming from the same supplier or source shall constitute a lot.

B-4.2 Sub-lot

For the purpose of sampling, all the wagons in a lot shall be divided into a suitable number of homogeneous sub-lots of approximately equal mass in acordance with Table 5.

B-4.3 A minimum of 25 percent of wagons shall be selected at random from the sub-lot. The corresponding number of increments to be taken from

a sub-lot shall be distributed over the selected wagons with a view to determining the number of increments that should be taken from each of the selected wagons in the sub-lot.

Table 5 Scale of Sampling for Bulk and Wagons
(*Clauses* B-3.2, B-3.3 *and* B-4.2)

Material in the Bulk	No. of Sub-lots	No. of Increments from Each Sub-lot
Up to 200 tonnes	2	20
201 to 500 tonnes	3	30
501 to 2 000 tonnes	4	40
2 001 tonnes and over	5	50

B-4.4 All the increments taken from the selected wagons in a sub-lot shall be pooled together to constitute a gross sample.

B-5 PREPARATION OF SAMPLE

B-5.1 All the increments from the same sub-lot shall be thoroughly mixed to constitute a gross sample representing the sub-lot. The minimum size of the gross sample shall be about 3 kg. If the gross sample is less than this, additional number of increments shall be drawn so as to make up the required quantity.

B-5.2 The gross sample shall be suitably reduced by the method of coning and quartering or any other suitable method to about 750 g which shall be divided into three portions called laboratory samples, one for the purchaser, another for the supplier and the third as the referee sample, each duly placed in the sample container.

B-6 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

B-6.1 Number of Tests

One laboratory sample from each sub-lot shall be tested individually for all the requirements of this specification and the sub-lot shall be considered satisfactory if the laboratory sample satisfies all the requirements of this specification.

B-6.2 Criteria for Conformity

The lot shall be declared to conform with the requirements of this specification if all the sub-lots (*see* **B-6.1**) are found satisfactory.

ANNEX C

(Foreword)

COMMITTEE COMPOSITION

Ceramicware Sectional Committee, CHD 9

Organization	*Representative(s)*
Central Glass & Ceramic Research Institute, Ahmedabad	DR K. N. MAITI (***Chairman***)
All India Pottery Manufacturers Association, Kolkata	SHRI S. K. GHOSH SHRI A. K. JOSHI (*Alternate*)
Bharat Heavy Electricals Limited, Bangalore	SHRI A. M. VAIDYA SHRI B. N. SHIVANANDA (*Alternate*)
Central Glass & Ceramic Research Institute, Kolkata	SHRI S. CHAKRABARTI
Chemicals & Allied Products Export Promotion Council, Kolkata	SHRI B. D. KOTHARI SHRI VED KAPOOR (*Alternate*)
Controller of Quality Assurance, Kanpur	SHRI A. K. BANDYOPADHYAY SHRI R. M. GAUTAM (*Alternate*)
Development Commissioner (SSI), New Delhi	SHRI S. K. KAPOOR SHRI A. S. SOOD (*Alternate*)
E.I.D. Parry (India) Limited, Chennai	SHRI R. KARTIKEYAN SHRI A. K. MISHRA (*Alternate*)
Excel Frits & Colours Limited, Kolkata	SHRI G. M. AGARWAL SHRI R. K. CHOWDHURY (*Alternate*)
Export Inspection Council of India, New Delhi	SHRI KARAM CHAND SHRI B. BAHADUR (*Alternate*)
Geological Survey of India, Kolkata	SHRI INDRANIL RAY
H.R. Johnsons India Limited, Mumbai	SHRI S. G. HEGDE SHRI G. S. PATNAIK (*Alternate*)
Hindustan Sanitaryware and Industries Limited, Bahadurgarh	SHRI R. K. SOMANY SHRI SANDIP SOMANY (*Alternate*)
Hitkari Potteries Limited, Faridabad	SHRI SATISH MALHAUTRA SHRI AJAY KAPOOR (*Alternate*)
Hotel & Restaurant Association of India, New Delhi	SHRIMATI MALINI RAJENDRAN
Indian Institute of Ceramics, Kolkata	DR S. DASGUPTA KM RITU PARNA SEN (*Alternate*)
Industries Commissioner, Government of Gujarat, Gandhi Nagar	SHRI R. J. SHAH
Madhusudan Ceramics Ltd, Mehsana	SHRI G. K. LOYA SHRI S. K. GHATAK (*Alternate*)
Ministry of Defence (Defence Materials and Stores Research & Development Establishment), Kanpur	DR NIRBHAY SINGH SHRI S. B. YADAV (*Alternate*)
Ministry of Industry, Department of Industrial Development, New Delhi	SHRI P. K. JAIN DR K. R. MURTY (*Alternate*)
National Test House, Kolkata	SHRI B. K. BISWAS SHRI N. N. TARACHANDRANI (*Alternate*)
Porcelain Enameller's Association, Kolkata	SHRI B. S. GANGULI SHRI N. ROY (*Alternate*)
Rajasthan State Mineral Development Corporation Limited, Jaipur	SHRI S. K. PATNI
Research, Designs & Standards Organization, Lucknow	DSC (Stds) DSC-SS
Welcome Group, Maurya Sheraton Hotel, New Delhi	REPRESENTATIVE
West Bengal Ceramic Development Corporation Limited, Kolkata	SHRI A. K. ROY SHRI TAPAS DUTTA (*Alternate*)
BIS Directorate General	SHRI S. K. CHAUDHURI, Director & Head (Chem) [Representing Director General (*Ex-officio*)]

Member Secretary
SHRI N. K. BANSAL
Joint Director (Chem), BIS

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc : No. WRD 3 (281).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Telegrams : Manaksanstha
(Common to all offices)

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Northern	: SCO 335-336, Sector 34-A, CHANDIGARH 160 022	60 38 43 60 20 25
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600 113	254 12 16, 254 14 42 254 25 19, 254 13 15
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400 093	832 92 95, 832 78 58 832 78 91, 832 78 92

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